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RESEARCH ARTICLE

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Successful plant growth in acid mine drainage-impacted soil using pot-based experiments with waste amendments

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Abstract

This paper addresses the challenge of remediating soil impacted by acid mine drainage (AMD) using an innovative and sustainable Technosol-based approach to stabilize soil and facilitate vegetation recovery. The study assessed the effectiveness of Technosols made from recycled organic (water clarification sludge) and inorganic (siderurgical slags and red gypsum) wastes in mitigating the detrimental effects of AMD on soil properties, pore water chemistry, and plant growth through a 4-month pot experiment. Technosols significantly improved soil health by neutralizing net acidity (296 mmol H^+ kg^{-1}), raising pH levels from extremely acidic (3.3) to mildly alkaline (7.7–8.0), and limiting the mobility of potentially toxic elements (PTEs). Dissolved Cu and Zn concentrations dropped from 80.21 and 72.08 mg L^{-1}, respectively, to below 1 mg L^{-1} by the end of the monitoring period. The experiment identified several concomitant mechanisms of PTE retention, such as decreased dissolution of metal-bearing minerals, precipitation reactions and adsorption onto Fe and Al (oxy)hydroxides. Aqueous speciation modelling indicated a decline in toxic metal forms (e.g. Al^{3+}, $AlSO_4^+$, Cu^{2+}, Zn^{2+} and $H_2AsO_4^-$) in soil pore water after treatment, thus reducing phytotoxicity. Additionally, waste amendments enhanced nutrient availability, with nitrate concentrations reaching up to 417 mg L^{-1}, supporting seed germination and seedling establishment. The most effective Technosol, combining water treatment sludge and white steel slag (60:40 w/w), enabled robust growth of *Brassica juncea*. Principal component analysis showed a strong correlation between healthy plant responses (survival rate, plant height, leaf number, biomass production) and improved soil pore water parameters (pH, PTEs, aluminium, calcium, bicarbonate and nitrate ions), highlighting the benefits of waste amendments. These findings underscore the potential of waste-derived Technosols in stabilizing AMD-impacted soils and promoting thriving plant growth. However, further validation in field trials with diverse plant species is recommended for real-world applications.

KEYWORDS
Brassica juncea, ecological restoration, non-hazardous wastes, soil reclamation, Technosol, Tharsis mines

1 | INTRODUCTION

Acid mine drainage (AMD) poses a significant environmental challenge arising from the exposure of sulphide minerals, notably pyrite, to air and water (Blowes et al., 2003; Nordstrom, 2011). Sulphide oxidative weathering leads to acid generation and the release of potentially toxic elements (PTEs) into the surrounding environment. This process raises substantial concerns in abandoned mine sites (Fernández-Caliani et al., 2009, 2019; Madejón et al., 2011, 2021), causing severe soil degradation, hindering plant growth, and posing health risks. AMD-affected soils often lose their natural resilience and capacity to immobilize harmful contaminants, becoming sources of acidic leachates with high levels of iron, sulphate and PTEs (Fernández-Landero et al., 2023). Sustainable remediation strategies are essential to mitigate contaminant dispersion and protect ecosystems and human health (Niu & Lin, 2021) in these environmental significant soils.

Remediating AMD-impacted soils is a complex, site-specific process that requires effective techniques for chemical stabilization and ecological restoration. In situ treatment involves using soil amendments to reduce contaminant leachability and bioavailability (Adriano et al., 2004; Bolan et al., 2014; Fernández-Caliani et al., 2021; Kumpiene et al., 2008; Palansooriya et al., 2020; Uchimiya et al., 2010). Common amendments for acidic, metal-contaminated soils include liming materials combined with organic additives (compost, biochar or manure) to improve soil fertility, enhance water holding capacity and stimulate microbial activity.

In recent years, Technosols (technogenic soils) have emerged as a promising alternative to traditional post-mining reclamation methods (Aguilar-Garrido et al., 2023; Ahirwal & Maiti, 2018; Arán et al., 2022; Asensio et al., 2013; Fernández-Caliani et al., 2024; Santos et al., 2019; Watkinson et al., 2017). This innovative approach involves blending non-hazardous waste materials or by-products with contaminated soil to create environments conducive to vegetation recovery and no-food biomass production. The benefits include cost-effectiveness, reduced resource reliance and waste reuse, aligning with circular economy principles (Ruiz et al., 2020; Séré et al., 2008; Watkinson et al., 2017; Weiler et al., 2020).

After achieving soil stabilization, ecological restoration becomes feasible, playing a crucial role in maintaining ecological balance and preventing further erosion. Selecting plant species depends on various factors like soil conditions, hydrology and climate, especially in AMD-impacted regions where species resilient to high PTE levels are needed (Barba-Brioso et al., 2023; Tordoff et al., 2000). Certain metal-tolerant plants, such as Indian mustard (*Brassica juncea* L.), are valuable for phytostabilization and soil revegetation (Forján et al., 2018; Lai et al., 2008; Novo & González, 2014; Pérez-Esteban et al., 2014) due to their ability to absorb and accumulate PTEs without succumbing to toxicity. Moreover, *B. juncea* serves as a reliable indicator of metal phytoavailability (Clemente et al., 2005), aiding in the assessment of remediation effectiveness.

This study explores the potential of using strategic combinations of non-hazardous waste materials to create sustainable Technosols for supporting plant growth in AMD-degraded landscapes. Through controlled pot-scale experiments, the research aimed to (1) elucidate the specific challenges associated with AMD-impacted soils; (2) evaluate the effectiveness of waste amendments in mitigating the adverse effects of AMD and (3) unveil how Technosols influence the establishment of *B. juncea* in remediated soil and the capacity to support healthy vegetation growth. By addressing these objectives, the study seeks to contribute to the development of sustainable practices for reclaiming AMD-affected soils and promoting broader ecological restoration efforts in post-mining landscapes.

2 | MATERIALS AND METHODS

2.1 | Mine soil and wastes used for Technosol construction

Soil samples were randomly collected from three locations within a barren area severely affected by AMD discharges (Fernández-Caliani et al., 2022; Fernández-Caliani & Barba-Brioso, 2010; Madejón et al., 2011) in the Tharsis mining district, Spain (Figure 1). Composite samples from the topsoil layer (0–20 cm) were air-dried, gently disaggregated and sieved to 2 mm. A portion of the sieved sample was then finely ground and passed through a 63 μm sieve for subsequent chemical analysis.

The AMD-affected soil was analysed for particle size, pH, redox potential (Eh), electrical conductivity, mineral composition, carbon and sulphur content, nitrogen and phosphorous levels, cation exchange capacity, exchangeable cations and total concentrations of major and PTEs. Acid soil drainage prediction was accomplished using an acid–base accounting approach, which assesses the total acid generating and neutralizing capacities through the acid–base accounting equation (Ahern et al., 2004): Net acid generation (NAG) = potential sulphidic acidity (PSA) + titratable actual acidity (TAA) + retained acidity − acid neutralizing capacity.

Four locally sourced waste materials were selected for Technosol construction: (1) water clarification sludge (WCS), (2) white slag from steel production (WSS), (3) furnace slag from iron production (FIS) and (4) red gypsum (RGY), a by-product of titanium dioxide pigment production. These materials, classified as non-hazardous wastes according to the European Waste Catalogue (Commission Decision 2000/532/EC), served specific roles in Technosol formulation, with WCS contributing organic nutrients (Dassanayake et al., 2015), WSS and FIS acting as liming agents (Yang et al., 2022) and RGY potentially improving soil structure while reducing contaminant mobilization (Peacock & Rimmer, 2000; Rodríguez-Jordá et al., 2010). These materials are currently landfilled 60 km from the reclamation site.

2.2 | Experimental setup

The experimental setup aimed to tailor waste amendments to reclaim AMD-impacted soils and promote vegetation restoration. Waste materials were characterized for specific surface area, pH, Eh,

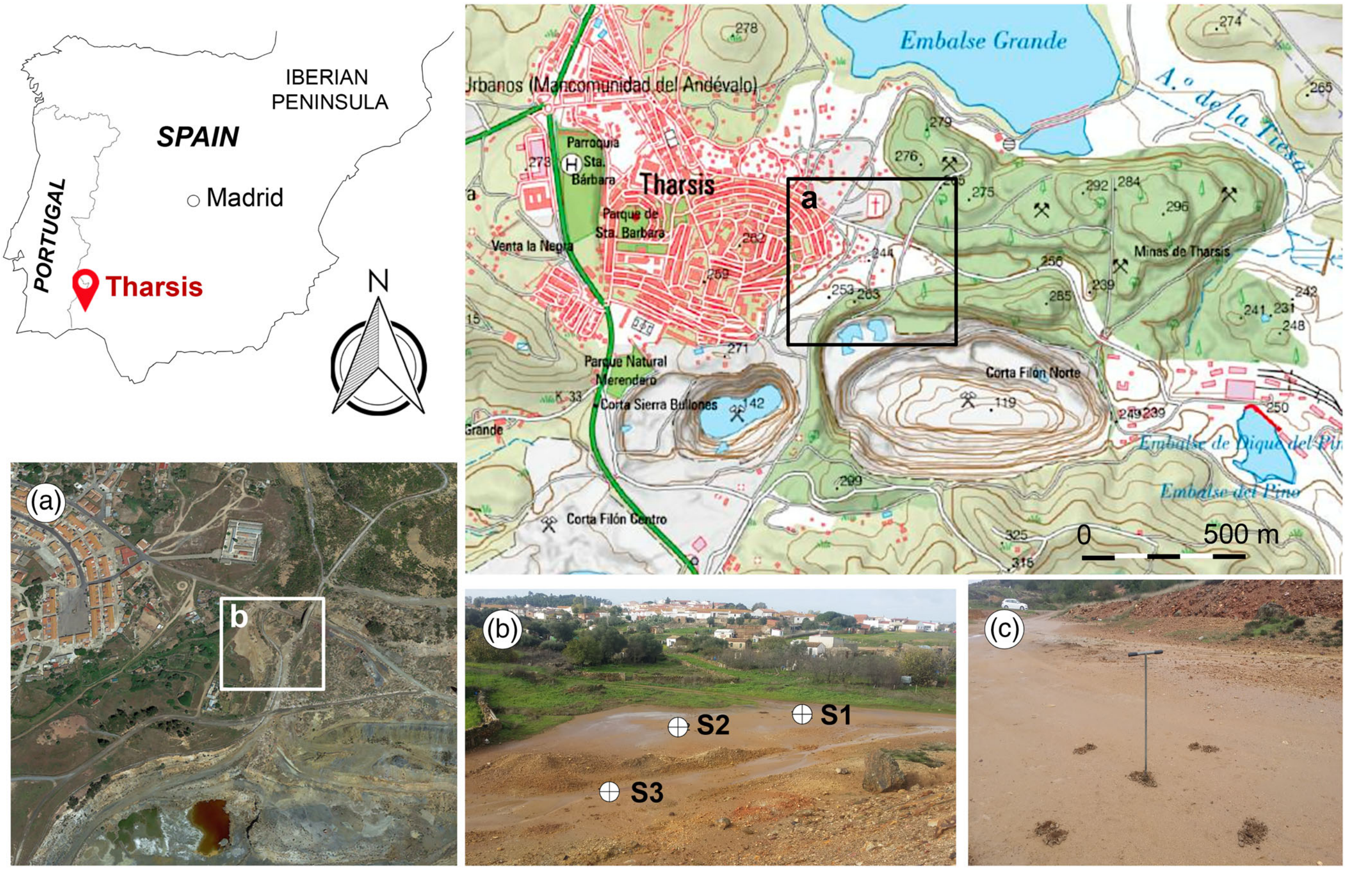


FIGURE 1 Location map of the Tharsis mining area showing: (a) an aerial view of the study area, (b) the sampling sites and (c) the soil sampling technique using a hand auger. [Colour figure can be viewed at wileyonlinelibrary.com]

TABLE 1 Composition of Technosols derived from water clarification sludge (WCS), white steel slag (WSS), furnace iron slag (FIS), and red gypsum (RGY).

Technosol	Technosol composition		Waste amendment	Amendment composition		
Control	100% mine soil (S3)		–	–		
S3T2-10	90% S3	10% WA1	WA1	60% WCS	20% WSS	20% RGY
S3T2-25	75% S3	25% WA1	WA1	60% WCS	20% WSS	20% RGY
S3T2-50	50% S3	50% WA1	WA1	60% WCS	20% WSS	20% RGY
S3T5-25	75% S3	25% WA2	WA2	60% WCS	20% FIS	20% RGY
S3T5-40	60% S3	40% WA2	WA2	60% WCS	20% FIS	20% RGY
S3T5-50	50% S3	50% WA2	WA2	60% WCS	20% FIS	20% RGY
S3T8-5	95% S3	5% WA3	WA3	60% WCS	40% WSS	–
S3T8-25	75% S3	25% WA3	WA3	60% WCS	40% WSS	–
S3T8-50	50% S3	50% WA3	WA3	60% WCS	40% WSS	–
S3T11-25	75% S3	25% WA4	WA4	60% WCS	40% FIS	–
S3T11-35	65% S3	35% WA4	WA4	60% WCS	40% FIS	–
S3T11-50	50% S3	50% WA4	WA4	60% WCS	40% FIS	–

Note: Mixing percentages are expressed in weight.

electrical conductivity, carbon content, nutrient levels and total concentrations of major and PTEs.

Soil amendments involved blending WCS, WSS, FIS and RGY waste materials in a 60:40 (w/w) ratio to achieve a balanced mixture of organic and inorganic components (Asensio et al., 2019; Santos et al., 2016; Yao et al., 2009). The waste amendments were mixed with mine soil at various rates (Table 1), with untreated mine soil (sample S3) serving as the control. The resulting Technosols underwent leaching tests to assess leachability and guide optimal blend selection for planting.

Selected planting blends were homogenized, moistened with distilled water and equilibrated for 14 days at room temperature before being transferred to pots (15 cm depth and diameter) and planted with *B. juncea* for 4 months. Germination assays in 90 mm Petri dishes confirmed the viability of *B. juncea* seeds. The dishes were kept at room temperature under a 10–14 h day/night photoperiod and monitored daily for germination progress. Five seedlings with expanded cotyledons were transplanted into Technosol pots. Each treatment had three replicates, including negative (mine soil) and positive (mulch) controls. The pots were placed in a glasshouse environment with controlled temperature, humidity and light exposure, while regular irrigation was applied to maintain optimal soil moisture levels.

A 4-month pot trial monitored the soil solution chemistry in both Technosol and control pots. Soil solution was sampled monthly at maximum water holding capacity using Rhizon samplers (Clemente et al., 2008). The extracted solution was analysed for pH, electrical conductivity and total concentrations of major elements and PTEs. Plant health, growth and development were evaluated by monitoring survival rate, height, leaf number, flowering time and biomass production.

2.3 | Analytical procedures

Phase identification was conducted by X-ray diffraction (XRD) on a BRUKER D8-Advance diffractometer, using monochromatic CuKα radiation (40 kV, 30 mA). Selected particles were examined using field emission scanning electron microscopy (FESEM) with a JEOL JSM-IT500HR instrument operating at 20 kV, and were chemically analysed with an energy-dispersive X-ray spectrometer coupled to the FESEM. Grain-size distribution and specific surface area were determined using a Mastersizer 3000 laser diffraction analyser. For pH, Eh and electrical conductivity measurements, a 10 g soil sample was mixed with 25 mL of deionized water. Additional pH measurements were performed in 1 M KCl (1:2.5 m/v) and 30% H_2O_2 (1:20 m/v) suspensions, with H_2O_2 added until bubbling ceased (Urrutia et al., 1992).

PSA was determined by extracting pyrite sulphur (S_{pyr}) with hot 6 M HNO_3, following the ASTM D2492-02 standard test method for sulphate removal prior to pyrite extraction. The extracted S_{pyr} was quantified by inductively coupled plasma optical emission spectroscopy (ICP-OES) with an Agilent 5110 instrument and converted to PSA assuming complete pyrite oxidation, which releases two moles of protons per mole (Dold, 2017; Nordstrom, 1982).

TAA measured readily available acidity in the soil, considering soluble sulphates and exchangeable acid cations. This was achieved by titrating a 1 M KCl soil suspension with 0.1 M NaOH until reaching a pH endpoint of 7.0 (McElnea et al., 2002). The acidity retained in sparingly soluble hydroxy-sulphate minerals, like jarosite, was estimated by quantifying extractable sulphate sulphur (S_{jar}) using 0.2 M NH_4-oxalate. The extracted S_{jar} was analysed by ICP-OES and converted to equivalent acidity units (mmol H^+ kg^{-1}) based on the assumption that one mole of sulphur in jarosite releases 1.5 moles of protons (Dold, 2003).

A static NAG test assessed the overall acid-forming potential of the mine soil. This test mimics pyrite oxidation by reacting the soil with a 30% H_2O_2 solution while monitoring pH variations. The solution is then titrated with 0.1 M NaOH until reaching a neutral pH, accounting for any acid neutralizing minerals present in the soil (Monterroso & Macías, 1998).

Total carbon and nitrogen were determined via dry combustion using an ELTRA CHS-580A analyser. Plant-available phosphorus and assimilable potassium were extracted with 0.5 N $NaHCO_3$ (Olsen & Sommers, 1982) and 0.01 M $CaCl_2$ (Houba et al., 1996) respectively. Effective cation exchange capacity was assessed by leaching the soil with a 1 M NH_4Cl solution. The extracted base cations were analysed using flame atomic absorption spectroscopy, while exchangeable acid cations were measured through titration with 0.01 N NaOH.

Major and trace element contents in mine soil and waste samples were analysed by ICP-OES following a four-acid digestion method. Quality control measures ensured data accuracy, including reagent blanks, duplicates and certified reference materials (OREAS series). Element concentrations deviated from reference values by less than 10% relative standard deviation (RSD), with most elements showing excellent reproducibility (RSD below 5%).

A standardized leaching test (EN-12457-4) evaluated PTE mobilization from Technosols. Leachates were filtered (0.45 μm pore size), acidified, and analysed for major elements by ICP-OES, while PTE concentrations were measured by ICP mass spectrometry (Agilent 7700 ICP-MS). Quality control measures, including external calibration, replicates, blanks and analysis of SRM 1640a, ensured outstanding accuracy (SRM recovery within 5%) and precision (RSD below 10%). Nitrate, fluoride and chloride concentrations were determined using ion chromatography (Metrohm 883 Basic IC plus instrument), while bicarbonate content was measured via titration with 0.02 M HCl.

2.4 | Statistical analysis and geochemical modelling

Statistical analysis was performed using SPSS 27.0 to assess Technosol influence on soil pore water chemistry. Left-censored data were imputed as half of the detection limit, and replicate variability was quantified by standard errors. Differences between Technosol-based treatments and controls were assessed using Student's *t*-test and one-way ANOVA, preceded by Levene's test to confirm homogeneity of variance. All tests adopted a significance level of $p < 0.05$. Principal component analysis (PCA) explored relationships between soil pore water chemistry and plant responses.

The PHREEQC chemical equilibrium modelling software (Parkhurst & Appelo, 2013) was employed to predict the aqueous speciation of PTEs and determine mineral saturation states using Equation (1):

$$SI = \log(IAP/K_s) \quad (1)$$

where SI is the saturation index, IAP denotes the ion activity product and K_s represents the solubility product constant. A SI value of 0 indicates equilibrium solubility, positive values suggest oversaturation and potential precipitation, while negative values imply undersaturation and potential dissolution.

3 | RESULTS AND DISCUSSION

3.1 | Properties and constituents of the mine soil

The AMD-impacted soil exhibited loam texture, extremely acidic reaction (pH_{H_2O} 2.7–3.4), strongly oxidizing conditions (Eh = 706 mV) and moderate salinity. Similar pH values were obtained using a 1 M KCl solution, suggesting potential constraints for plant growth and microbial activity. XRD analysis identified quartz, mica, kaolinite and jarosite as the dominant soil minerals, with minor feldspars, gypsum and hematite. Importantly, the absence of carbonates indicates limited buffering capacity against further acidification.

The observed $pH_{H_2O_2}$ values aligned with the low S_{pyr} content (0.10%), resulting in relatively low PSA (65 mmol H^+ kg^{-1}) linked to the oxidation of mineral dust particles derived from nearby mine waste dumps (Chopin et al., 2003; Fernández-Caliani et al., 2013; Fernández-Caliani & Barba-Brioso, 2010). Low TAA values (57–87 mmol H^+ kg^{-1}) imply a limited capacity of the soil to generate acidity through the dissolution of readily soluble sulphate minerals and the release of acid cations from exchange sites.

Considerable differences between S_{total} and S_{pyr} levels denote the occurrence of sulphate minerals, primarily jarosite, as confirmed by XRD analysis. S_{jar} was the dominant form of sulphur, ranging from 1.70% to 2.53%, representing retained acidity released through slow jarosite hydrolysis, estimated at 797–1186 mmol H^+ kg^{-1}. This finding underscores the significance of jarosite as the main reservoir of acidity, accounting for 86%–90% of the total acid-generating capacity, and its role in predicting long-term acidification trends.

The H_2O_2-based static test revealed a mean NAG value of 296 mmol H^+ kg^{-1}, which reflects the net acid production, assuming simultaneous acid generation and acid neutralization reactions (Karlsson et al., 2018; Miller et al., 1997). This result is consistent with those from nearby Rio Tinto mine soils (Fernández-Landero et al., 2023), highlighting persistent acidity and limited acid neutralizing potential.

Mine soil analysis showed severe limitations in essential plant nutrients, including nitrogen (0.32%) and plant-available phosphorus (P_{Olsen} = 0.014%), aligning with observations from other regional mine soils (Alvarenga et al., 2022). The high prevalence of exchangeable acid cations (over 52% of effective cation exchange capacity) can further exacerbate nutrient deficiencies for plant uptake. Organic carbon content varied widely (0.2%–3.7%), with the higher values being likely due to the presence of carbonaceous shale fragments derived from nearby mine waste dumps.

As shown in Table 2, the AMD-impacted soil showed a distinctive geochemical signature characterized by relatively high levels of Fe (9.6%–11.0%) and Al (6.9%–7.3%), with smaller contributions from K and S. This elemental profile aligns with the mineralogical composition of the mine soil, reflecting the occurrence of jarosite, iron oxides and aluminosilicate minerals. A concerning aspect of the AMD impact was the consistently elevated levels of PTEs. Total concentrations of Pb (up to 1610 mg kg^{-1}), Cu (up to 942 mg kg^{-1}) and As (up to 783 mg kg^{-1}) exceeded regional baseline levels (Galán et al., 2008; Martín-Méndez et al., 2023) by one to two orders of magnitude, indicating severe multi-elemental contamination. Of particular concern is the surpassing of As concentrations beyond the screening level (40 mg kg^{-1}) set for industrial land use in southern Spain (Junta de Andalucía, 2015), indicating an unacceptable risk to potential receptors.

3.2 | Characterization of waste materials used as amendments

The tested waste materials (WCS, WSS, FIS and RGY) displayed diverse characteristics across particle size distribution, pH, electrical conductivity, agronomic properties and element concentrations (Table 3), reflecting their varied origins and compositions.

WCS exhibited a slightly acidic to near-neutral pH, low electrical conductivity, and a high organic carbon content (11.50%), making it suitable as an organic fertilizer. It boasts significant amounts of available phosphorus (16.7 mg kg^{-1}) and potassium (57.7 mg kg^{-1}), providing a source of essential nutrients. The observed high Al content (>10%) is attributed to aluminosilicate clay minerals and the use of Al-based coagulants during drinking water treatment. XRD analysis further revealed a diverse mineral assemblage encompassing quartz, feldspars, iron oxides and gypsum.

WSS showed exceptional alkalinity (pH_{H_2O} = 12.4), indicating its potential to counteract soil acidity, along with high electrical conductivity (7.27 mS cm^{-1}). FESEM-energy-dispersive X-ray spectrometer analysis revealed distinct calcium silicate phases, calcite and Mg oxides and aluminates. Despite lacking organic carbon, WSS compensates with remarkable levels of plant-available phosphorus (22.8 mg kg^{-1}) and potassium (151 mg kg^{-1}), serving as a valuable nutrient source. High contents of Ca (>10%) and Mg (6.67%) further enhance its potential as an alkaline soil amendment.

FIS displayed an alkaline pH_{H_2O} (10.0) and low electrical conductivity. It showed 1.70% total carbon, with 1.00% being organic carbon, and low total nitrogen content (0.09%). Moderate levels of available phosphorus (18.7 mg kg^{-1}) and potassium (56 mg kg^{-1}) further enhanced its potential as a soil amendment. Additionally, it exhibited a relatively high Fe content (7.56%) ascribed to iron-silicate phases. XRD analysis identified abundant quartz with minor anorthite and calcite.

RGY had a slightly alkaline pH_{H_2O} (7.76) and moderate electrical conductivity. It is composed mainly of gypsum with minor iron oxides, rutile, ilmenite and zircon. Despite its predominantly inorganic nature,

TABLE 2 Soil properties, acidity pools and chemical composition of mine soil samples.

Parameter	Mine soil samples (<2 mm fraction) S1	S2	S3	Mean	Std. dev.
Color (dry)	10YR 6/4	10YR 6/3	10YR 6/4	–	–
Sand (%)	51.6	30.9	41.4	41.3	10.4
Silt (%)	41.4	56.9	47.1	48.5	7.8
Clay (%)	7.0	12.2	11.5	10.2	2.8
Electrical conductivity (mS cm^{-1})	3.59	1.94	2.02	2.52	0.93
Eh (mV)	656	694	767	706	56
pH_{H_2O}	3.4	3.2	2.7	3.09	0.39
pH_{KCl}	3.3	3.0	2.4	2.92	0.46
pH_{Ox}	2.7	2.6	2.3	2.5	0.2
Acidity (mmol H^+ kg^{-1})					
Potential sulphidic acidity	81	69	44	65	19
Titratable actual acidity	57	64	87	69	16
Retained acidity	1073	1186	797	1019	200
Total acid generation	1211	1319	928	1153	202
Net acid generation	417	281	189	296	115
S_{jar} (%)	2.29	2.53	1.70	2.17	0.43
S_{py} (%)	0.13	0.11	0.11	0.10	0.03
C_{inorg} (%)	0.0	0.1	0.2	0.1	0.1
C_{org} (%)	3.7	1.8	0.2	1.9	1.7
N_{total} (%)	0.50	0.34	0.10	0.32	0.20
P_{Olsen} (%)	0.027	0.013	0.001	0.014	0.013
Exchangeable cations ($cmol^+$ kg^{-1})					
Ca^{2+}	6.9	11.1	10.8	9.6	2.3
Mg^{2+}	4.7	3.2	3.0	3.6	0.9
Na^+	0.2	0.1	n.d.	0.1	0.1
K^+	n.d.	n.d.	n.d.	n.d.	n.d.
$H^+ + Al^{3+}$	19.4	14.3	10.6	14.8	4.4
Cation exchange capacity ($cmol^+$ kg^{-1})	31.2	28.8	24.5	28.2	3.4
Non-acid cation saturation (%)	37.7	50.3	56.7	48.2	9.7
Major element concentrations (wt %)					
Al	6.91	7.26	6.90	7.02	0.21
Ca	0.22	0.26	0.15	0.21	0.06
Fe	10.5	11.0	9.60	10.37	0.71
K	1.76	2.48	2.73	2.32	0.50
Mg	0.44	0.45	0.34	0.41	0.06
Mn	0.019	0.017	0.018	0.018	0.001
Na	0.40	0.54	0.49	0.48	0.07
Ti	0.31	0.38	0.43	0.37	0.06
S	2.25	2.44	1.93	2.21	0.26
P	0.835	0.333	0.048	0.045	0.398
Trace element concentrations (mg kg^{-1})					
As	714	556	783	684	116
Bi	14	12	10	12	2
Cd	0.7	0.4	0.5	0.53	0.15
Co	13	11	10	11.3	1.5

TABLE 2 (Continued)

Parameter	Mine soil samples (<2 mm fraction)				
	S1	S2	S3	Mean	Std. dev.
Cr	103	102	87	97	9
Cu	942	540	339	607	307
Ni	22	24	19	21.7	2.5
Pb	1610	1010	1200	1273	307
Sb	86	27	<5	56	44
Tl	<5	<5	<5	<5	<5
Zn	190	136	111	146	40

Abbreviation: n.d., not detected.

it provided some nutrient value with 130 mg kg^{-1} of assimilable potassium and 3 mg kg^{-1} of plant-available phosphorus. High levels of Ca and S (>10% each) are attributed to the abundance of gypsum, while the significant Fe content (5.39%) stems from iron oxides.

Although the overall PTE concentrations in these waste materials are generally lower than those observed in the AMD-impacted soil and adhere to Spanish legislation for Technosol formulation (ITR, 2008), elevated levels of Cu (294 mg in FIS) and Zn (315 mg kg^{-1} in RGY and 252 mg in WSS) may require careful consideration.

3.3 | Leachate quality

The leaching test provided valuable insights into the acidity/alkalinity and pollutant release of both mine soil and the tested waste amendments (WA1, WA2, WA3 and WA4). All waste amendments yielded alkaline leachates (Figure 2a), with WA1 and WA3 showing the highest alkalinity due to the incorporation of WSS as a liming material. Electrical conductivity ranged from 0.66 to 2.60 mS cm^{-1}, with RGY-bearing wastes (WA1 and WA2) registering the highest values. Leachate solutions revealed varying concentrations of diverse anions (Figure 2b), with sulphate particularly elevated (around 12,000 mg kg^{-1}) in WA1 and WA2 due to their gypsum content. Chloride levels ranged from 324 to 838 mg kg^{-1}, while nitrate concentrations varied between 124 and 230 mg kg^{-1}.

A significant concern when utilizing waste-derived amendments in agriculture and landscaping is the potential release of high levels of PTEs into the environment (Siddique et al., 2010). However, leachate analysis showed negligible concentrations of dissolved PTEs in the amendments, typically below 0.1 mg kg^{-1}. Conversely, the AMD-impacted soil produced highly acidic leachate (pH 2.8) with concerning levels of Cu (39.54 mg kg^{-1}) and Zn (22.66 mg kg^{-1}), while other PTEs remained below 1 mg kg^{-1}. These results indicate minimal leaching risk of contaminants, complying with Spanish regulations for safe Technosol construction (ITR, 2008).

The Technosol treatments effectively reduced the leaching of PTEs from the mine soil, particularly for Cu and Zn. Leaching of other divalent metals (Ni, Cd and Pb) was minimized to negligible or undetectable levels (Figure 2c). However, there was a slight increase in the leaching of As and Sb attributed to reduced anion adsorption onto soil particles under alkaline conditions (Bradl, 2004; Carabante et al., 2009; Fan et al., 2020). Importantly, despite this increase, the concentrations of leached As and Sb remained well below regulatory limits for Technosol construction (2 mg kg^{-1} for As and 0.7 mg kg^{-1} for Sb).

3.4 | Technosol pore water chemistry

Dissolved pore water PTE concentrations are a crucial indicator of environmental risk, revealing the fraction readily available for plant uptake (Clemente et al., 2008; Nolan et al., 2003). To gain insights into this, interstitial water from Technosols S3T2-25, S3T8-5 and S3T11-35 was analysed over a 4-month pot trial.

All Technosols displayed significantly higher pH values compared to the untreated control soil ($p < 0.002$) throughout the experiment (Figure S1). Technosols amended with WSS (S3T2-25 and S3T8-5) experienced a rapid and sustained increase in pore water pH, shifting from extremely acidic to mildly alkaline or near-neutral conditions. Conversely, Technosol S3T11-35, amended with FIS, demonstrated a less pronounced effect, remaining moderately acidic. These findings suggest that WSS holds significant promise for neutralizing net acidity. The strongly oxidizing state (Eh 712 mV) of the mine soil solution displayed a noteworthy shift towards less oxidizing conditions upon amendment application. The shift was most pronounced in Technosols S3T2-25 and S3T8-5, which reached moderately oxidizing states. In contrast, the untreated control soil maintained its highly oxidizing character throughout the experiment. The electrical conductivity of the pore water in the soil control exhibited a steady decline, stabilizing between 6.0 and 8.1 mS cm^{-1}. This decrease reflects the depletion of readily soluble sulphate salts through irrigation water leaching. However, Technosol treatments maintained consistent electrical conductivity ranges throughout the study period.

Technosols showed differential effects on the major element concentrations in the pore water (Figure 3). Waste amendments significantly reduced the levels of Al and Fe compared to the control soil

TABLE 3 Properties and chemical composition of the waste materials used as ingredients in Technosols.

		Waste materials (<2 mm fraction)			
Parameter	Unit	WCS	WSS	FIS	RGY
Munsell colour (dry)	–	2.5Y 5/3	2.5Y 6/1	10YR 4/1	7.5YR 6/6
Specific surface area	$m^2\ g^{-1}$	0.598	0.640	0.474	0.756
Electrical conductivity	$mS\ cm^{-1}$	0.69 ± 0.07	7.27 ± 0.06	0.78 ± 0.04	4.33 ± 0.25
pH_{H_2O}	–	6.74 ± 0.21	12.44 ± 0.11	10.00 ± 0.13	7.76 ± 0.61
C_{total}	%	11.82 ± 0.31	1.48 ± 0.13	1.67 ± 0.63	0.84 ± 0.02
C_{inorg}	%	0.34 ± 0.04	1.48 ± 0.60	0.70 ± 0.06	0.53 ± 0.22
C_{org}	%	11.49 ± 0.31	n.d.	0.98 ± 0.51	0.31 ± 0.02
Organic matter	%	19.83 ± 0.53	n.d.	1.72 ± 0.71	0.52 ± 0.04
N_{total}	%	2.12	0.01	0.09	0.08
C/N	–	5.6	150	18.9	3.9
$P_{available}$	$mg\ kg^{-1}$	16.7	22.8	18.7	3.0
$K_{available}$	$mg\ kg^{-1}$	57.7 ± 4.2	151 ± 8.2	56.0 ± 0.2	130 ± 5.8
Major elements	Detection limit (wt %)				
Al	0.01	>10	3.79	0.97	0.45
Ca	0.01	0.18	>10	1.68	>10
Fe	0.01	1.34	0.93	7.56	5.39
K	0.01	0.06	0.02	0.09	0.02
Mg	0.01	0.16	6.67	0.83	0.50
Na	0.01	0.06	0.09	0.29	0.07
P	0.001	0.175	0.001	0.024	0.006
S	0.01	0.78	0.42	0.04	11.0
Ti	0.01	< 0.01	0.12	0.03	0.47
Trace elements	Detection limit ($mg\ kg^{-1}$)				
As	2	42	<2	26	4
Bi	2	<2	<2	<2	<2
Cd	0.5	<0.5	<0.5	<0.5	2
Co	1	3	1	5	8
Cr	1	14	121	73	69
Cu	1	84	42	294	41
Hg	1	2	<1	<1	<1
Ni	1	15	12	54	26
Pb	3	23	16	88	58
Tl	2	<2	<2	<2	<2
Zn	1	71	252	226	315

Abbreviations: FIS: furnace iron slag; n.d., not detected; RGY, red gypsum; WCS, water clarification sludge; WSS, white steel slag.

(p < 0.00001). Notably, Al concentration dropped dramatically from 1178 mg L^{-1} to practically undetectable levels, addressing a major toxicity concern in acid mine soils (Fernández-Caliani & Barba-Brioso, 2010). This improvement is likely due to the formation of Al hydroxide solid phases (Sánchez-España et al., 2011). This hypothesis is supported by saturation indices predicted by PHREEQC modelling, which indicates a likelihood of precipitation for gibbsite (SI = 1.8–2.9), boehmite (SI = 1.5–2.6) and diaspore (SI = 3.1–4.3). These precipitates not only remove harmful Al from the solution but also provide additional binding sites for other contaminants, boosting the sorption capacity of the Technosols. Similarly, dissolved Fe concentrations decreased significantly from 10 mg L^{-1} to below 0.5 mg L^{-1}. Equilibrium modelling suggests this decrease is due to the formation of ferrihydrite (SI = 3.6–3.8), a mineral prevalent in ochreous sediments precipitated from Fe-rich waters with neutral to mildly alkaline pH (Bigham et al., 1992; Murad & Rojík, 2003).

All three amended soils displayed a significant increase (p < 0.00001) in dissolved Ca concentrations. This notable increase, particularly evident in Technosol S3T2-25 (peaking at 948 mg L^{-1}), can be attributed to the dissolution of calcite and calcium silicate

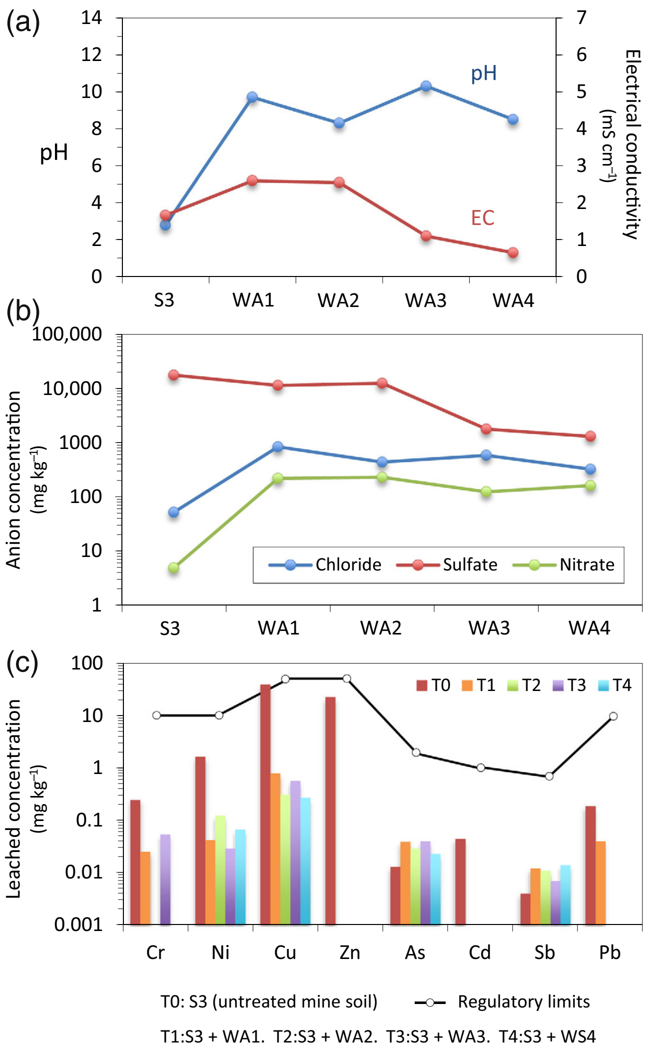


FIGURE 2 Effects of waste amendments on mine soil leachate composition: (a) pH and electrical conductivity of leachates from mine soil and waste amendments; (b) anion concentrations in leachates and (c) concentrations of potentially toxic trace elements leached from Technosols compared to regulatory limits for safe Technosol construction. S3, untreated mine soil; T, Technosol; WA, waste amendment. Composition details of waste amendments are provided in Table 1. [Colour figure can be viewed at wileyonlinelibrary.com]

compounds present in the waste amendments. Dissolved Mg concentration decreased significantly ($p < 0.01$) in Technosols S3T2-25 and S3T11-35, but increased ($p < 0.01$) in Technosol S3T8-5 due to its higher WSS content. Technosol S3T11-35 showed a noteworthy elevation in Na concentration compared to the control ($p < 0.00005$), whereas no significant changes were observed in Technosols S3T2-25 and S3T8-5 ($p > 0.05$). Overall, Technosol S3T11-35 proved most effective in promoting the solubilization of alkaline elements, while S3T8-5 displayed enhanced Mg mobilization.

As noted previously, waste amendments caused a pH increase, shifting towards moderately alkaline or near-neutral levels, while maintaining oxidizing conditions. This combined effect improved soil pore water quality by enhancing PTE retention through chemical

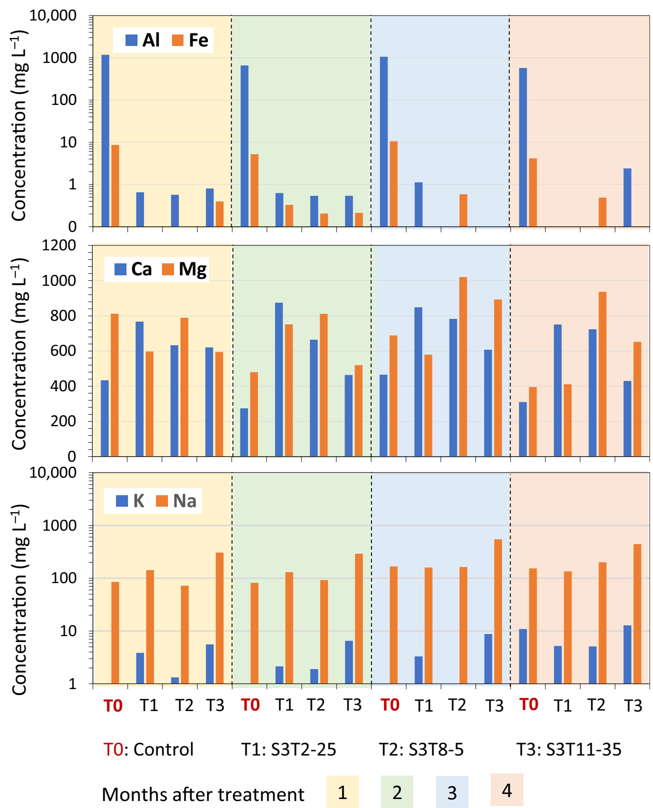


FIGURE 3 Comparative bar charts showing the temporal evolution of major element concentrations dissolved in the pore water extracts of the Technosols (T1, T2 and T3) and the control mine soil (T0) during the 4-month pot trial. [Colour figure can be viewed at wileyonlinelibrary.com]

stabilization (Basta & McGowen, 2004; Bolan et al., 2014; Kumpiene et al., 2008). Figure 4 illustrates the significant reduction in dissolved metal load within Technosols relative to the untreated (control) soil ($p < 0.00001$). In fact, treatments lowered mean concentrations of Cr, Co, Ni, Cu, Zn, Cd and Pb in pore water, with some declining by several orders of magnitude, clearly indicating reduced mobility and phytoavailability. Particularly, Cu and Zn concentrations dropped from initial levels of 80.21 and 72.08 mg L^{-1} to below 1 mg L^{-1} by the end of the monitoring period. The observed improvement in PTE immobilization can be ascribed to the alleviation of soil acidity, which reduces the activity of hydrogen ions in the soil solution, thereby decreasing competition for metal binding sites. This finding supports recent studies (e.g. Díaz et al., 2024; Paniagua-López et al., 2024) that demonstrate metal immobilization through soil amendment practices.

Waste amendment application in the mine soil triggered the precipitation of Cu-bearing phases, primarily copper ferrites. This is supported by positive saturation indices computed using PHREEQC (SI = 12.7–16.7). However, minerals containing Cd, Pb or Zn lacked positive SI values, suggesting that their mobility is likely controlled by adsorption processes onto newly formed phases rather than precipitation. Ferrihydrite may play a role in sequestering some of these PTEs through adsorption and co-precipitation mechanisms (Carlson et al., 2002; Michel et al., 2007; Sherman & Randall, 2003).

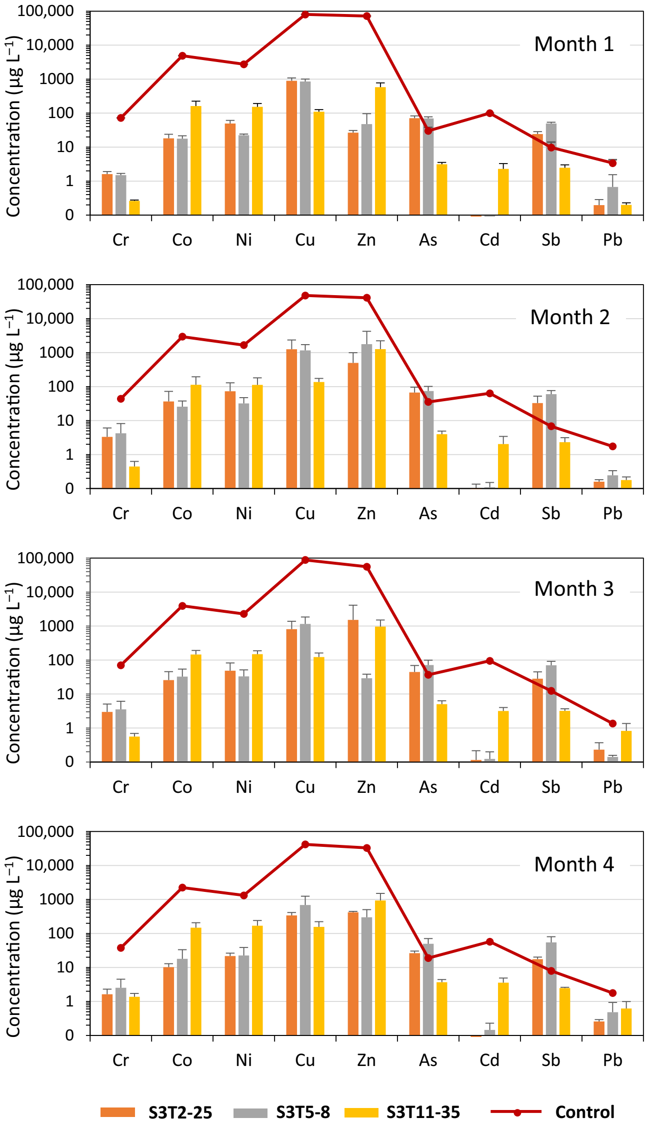


FIGURE 4 Comparative bar charts displaying trace element concentrations dissolved in the pore water extracts of the Technosols during the 4-month pot trial. Line charts show the trace element contents in the pore water of untreated mine soil (control). Error bars denote the standard deviation of the means ($n = 3$). [Colour figure can be viewed at wileyonlinelibrary.com]

Rising the pH to near-neutral and mildly alkaline levels in Technosols S3T2-25 y S3T8-5 increased the mobility of metalloids, specifically As and Sb, evidenced by their higher concentrations in pore water compared to the control ($p < 0.00003$). Despite concerns about potential As mobilization associated with the pH increase to the alkaline region (Hartley et al., 2004; Kumpiene et al., 2008), dissolved As concentrations remained below 50 µg L^{-1} in both Technosols after treatment. The enhanced mobility of As and Sb may result from an increase in negatively charged soil surface sites following acid neutralization (Bradl, 2004; Carabante et al., 2009), hindering the adsorption of harmful oxyanions. Conversely, Technosol S3T11-35, with an acidic soil solution, showed significant reductions in metalloid concentrations compared to the control ($p < 0.0001$), indicating its effectiveness as the most suitable treatment for all PTEs, including metalloids.

Sulphate was initially the predominant anion in the untreated mine soil solution, peaking at 23.04 g L^{-1}. This concentration decreased to 4.19 g L^{-1} over time due to the washing out of efflorescent sulphate salts formed on the soil surface. Upon treatment, mean chloride concentrations increased up to 1824 mg L^{-1} and nitrate concentrations up to 417 mg L^{-1}, exceeding untreated counterparts, which peaked at 376 mg L^{-1} and 61.2 respectively. Elevated nitrate levels benefit for plant growth as an essential nutrient. Bicarbonate also emerged as a prominent anion, peaking at 382 mg L^{-1} in Technosol S3T2-25.

Following the pot trial, sulphate remained the dominant anion in the mine soil solution (control), comprising 98% of the total anionic composition. However, Technosol treatments induced a shift in the species distribution pattern. Although sulphate remained significant (50%–66%), a new speciation model emerged (Figure S2), with chloride, bicarbonate and nitrate accounting for up to 31%, 28% and 25% respectively. This shift in the anionic balance can influence soil properties, affecting the mobility and speciation of contaminants.

Understanding the mobility, availability and toxicity of contaminants in the soil solution requires paying attention to their specific chemical forms rather than solely focusing on total dissolved concentration (Kalis et al., 2008). Through PHREEQC speciation calculations, insights into the distribution of PTEs among different aqueous species were gained by comparing untreated soil with Technosols after the 4-month pot trial (Figure 5).

In the untreated control soil solution, Al mainly formed harmful sulphate complexes, with the monomeric form ($AlSO_4^+$) comprising 83% of dissolved Al species. Notably, around 10% existed as highly toxic Al^{3+} ions, detrimental to plant growth (Chandra & Keshavkant, 2021). Waste amendment applications effectively mitigated this concern by promoting a shift towards less toxic hydroxylated species, such as $Al(OH)_4^-$ and $Al(OH)_2^+$. This shift, driven by the increased pH, reduces Al phytotoxicity by rendering it less soluble and more prone to form less harmful species (Bojórquez-Quintal et al., 2017).

Heavy metal speciation revealed a dominance of sulphate complexes in the mine soil solution, including neutral and negatively charged species ($CuSO_4$, $CdSO_4$, $Cd(SO_4)_2^{2-}$, $PbSO_4$, $ZnSO_4$ and $Zn(SO_4)_2^{2-}$). Moreover, a significant fraction of the dissolved metal load existed as free metal ions, posing a threat to plant growth through phytotoxicity (Kumar et al., 2021). Technosol application demonstrably altered this speciation pattern, leading to a greater diversity of metal species compared to the control. In Technosols S3T2-25 and S3T8-5, Cu speciation was dominated by $CuCO_3$ (77.5%–81.0%), creating a thriving environment for plants by reducing the activity of toxic Cu^{2+} ions. Zinc speciation was more complex, with a mixture of free metal, sulphate, carbonate and nitrate ions, while Cd primarily existed as free Cd^{2+}, along with sulphate complexes, and chloride ions. Changes in Cu and Zn speciation suggest a decrease in their mobility, although some free ions remained available to plants. While dissolved Pb and Cd concentrations decreased to

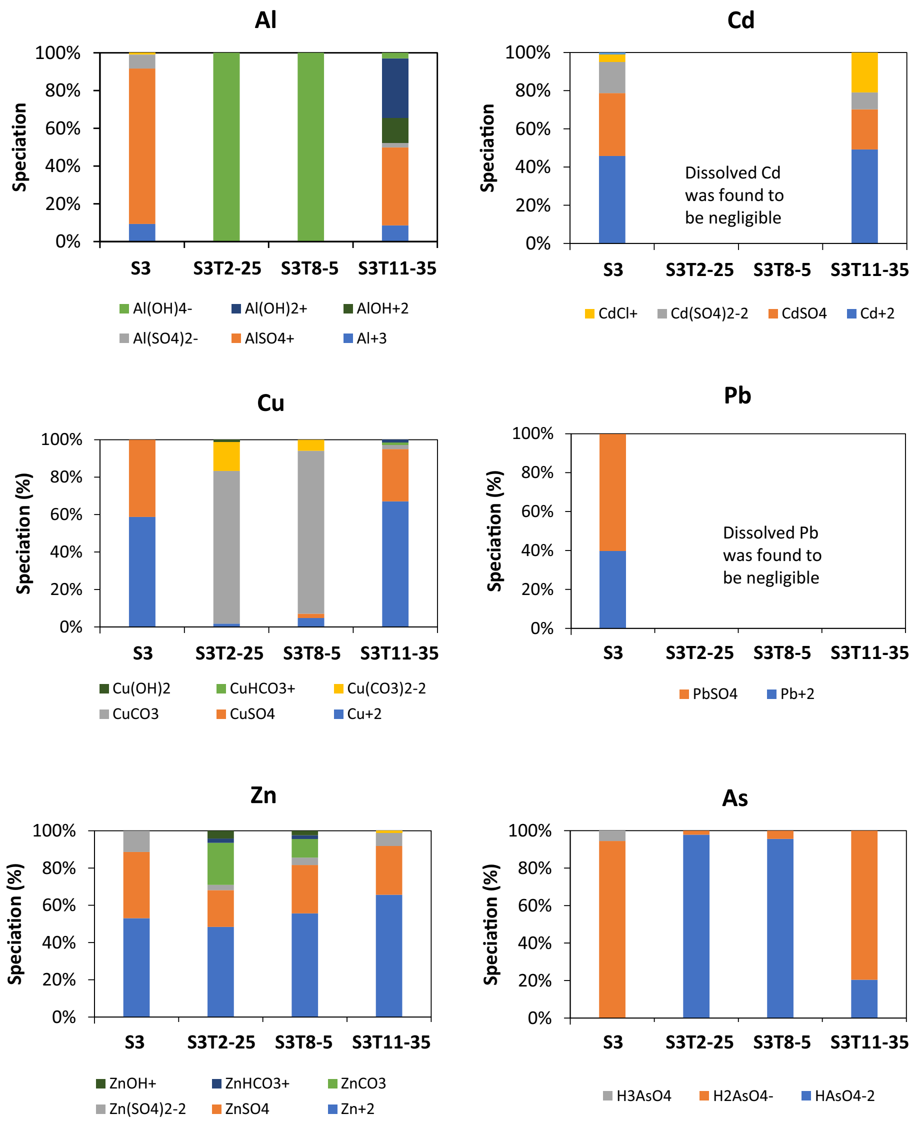


FIGURE 5 Aqueous speciation of trace elements of concern in the soil solution of Technosols (S3T2-25, S3T8-5 and S3T11-35) and control mine soil (sample S3) after the end of the pot trial, as predicted by PHREEQC geochemical modelling. [Colour figure can be viewed at wileyonlinelibrary.com]

negligible levels in Technosols S3T2-25 and S3T8-5, the presence of Cd^{2+} ions in Technosol S3T11-35 raises concerns regarding potential plant uptake.

Similar to other PTEs, the aqueous speciation of As in the amended soils shifted towards less harmful forms due to increased pH and oxygenation. Initially dominated by dihydrogen arsenate ($H_2AsO_4^-$), treatment induced a transition towards hydrogen arsenate ($HAsO_4^{2-}$), which is less mobile than H_3AsO_4 and $H_2AsO_4^-$ and much less toxic than arsenite species (Yamauchi & Fowler, 1994). Notably, Technosols S3T2-25 and S3T8-5 showed a remarkable dominance of this less harmful species, with 95%–99% of As existing as $HAsO_4^{2-}$.

3.5 | Effectiveness of waste amendments on plant growth

A seed germination assay using *B. juncea* revealed a significant positive effect of waste amendments on plant establishment in Technosols. The inherent limitations of the mine soils (strong acidity, nutrient deficiencies and high levels of dissolved PTEs) severely hampered germination. Conversely, Technosols enhanced both germination and early seedling growth, with seeds readily absorbing water, sprouting and developing cotyledons within a week. Germination was confirmed when the radicle reached 3 mm in length (ISTA, 2005). Germination rates varied across treatments (Figure 6a): untreated mine soil (0%), Technosol S3T11-35 (73.3%), Technosol S3T8-5 (80.0%), Technosol S3T2-25 (86.7%) and mulch control (90%).

Figure 7 depicts the 4-month progression of *B. juncea* plants in the pot trial. Although all seedlings successfully developed true leaves within a week, remarkable differences in plant establishment and growth were observed among treatments.

Technosol S3T2-25 demonstrated outstanding plant survival (100%), comparable to the mulched control (Figure 6b), indicating its effectiveness in fostering plant adaptation and resilience. Plants grew vigorously, reaching a maximum height of 59.4 cm with up to 51 leaves per pot by week 13 (Figure 6c,d). Technosol S3T8-5 maintained an 80% survival rate throughout the trial, with surviving plants

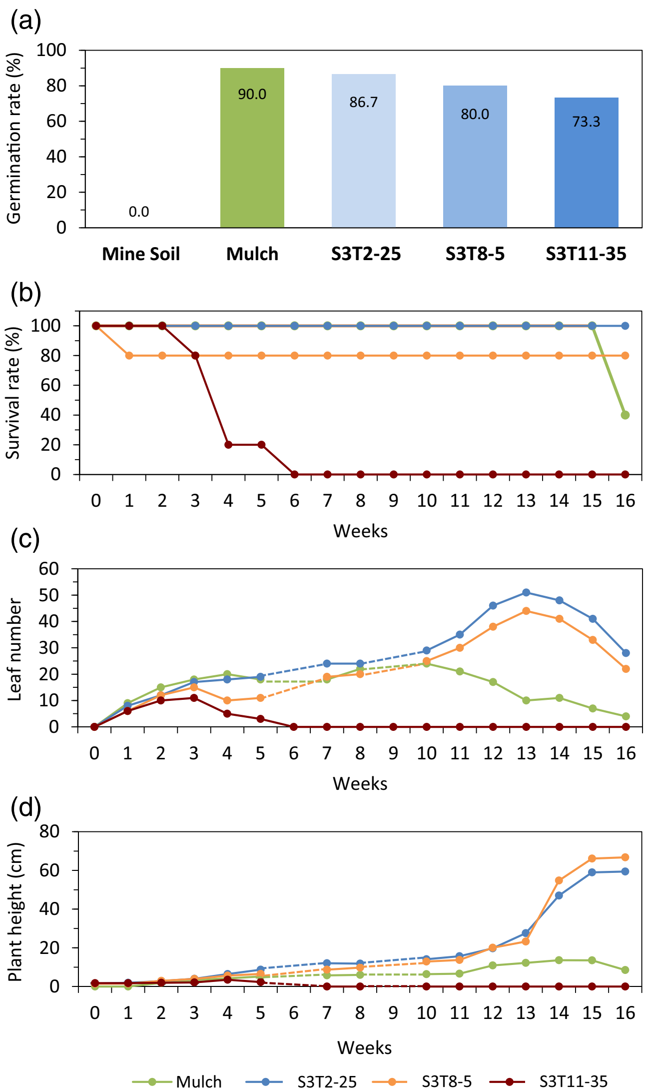


FIGURE 6 Bar chart comparing (a) seed germination rate of *Brassica juncea* in Technosols and mulch control, alongside line charts showing plant growth dynamics throughout the pot-based trial: (b) survival rate; (c) maximum plant height and (d) leaf number. [Colour figure can be viewed at wileyonlinelibrary.com]

achieving an even taller average height (66.8 cm) and a maximum leaf count of 44 by week 13. Notably, there were no statistically significant differences ($p > 0.053$) in plant growth parameters between the two Technosols, suggesting comparable effectiveness. Flowering commenced in both Technosols by week 12, mirroring the mulched control, with plants displaying vibrant yellow flowers and successfully completing seed production by week 13. At the conclusion of the trial, Technosol S3T2-25 yielded 6.734 g per pot of aboveground dry biomass, while Technosol S3T8-5 produced 5.574 g per pot.

In contrast, plant survival in Technosol S3T11-35 sharply declined, with only 20% survival by week 4 and complete mortality by week 6, indicating a limited capacity for *B. juncea* growth. The surviving plants displayed stunted growth, reaching an average peak height of just 3.5 cm by week 4, with limited leaf production (peaking at 11 leaves in week 3). Unlike the healthy growth observed in other Technosols, plants in Technosol S3T11-35 showed stress symptoms like limited growth, reduced leaf number, and foliar chlorosis, resembling effects typically associated with nutrient deficiency and metal stress (Chandra & Keshavkant, 2021; Kumar et al., 2021). By week 6, when all plants succumbed, cumulative aboveground dry biomass production was only 0.155 g.

PCA analysis elucidated the relationships between plant responses (survival rate, plant height, leaf number, biomass) and soil pore water variables (pH, trace elements, aluminium, calcium, bicarbonate, nitrate), providing further insights into how changes in soil solution chemistry influenced plant health and growth. The scores projected onto the two principal components (PC1 and PC2) delineated distinct clusters (Figure 8). PC1 explained 87% of the total variance and was driven by strong positive loadings for healthy plant responses and favourable soil conditions. High positive PC1 scores correlated with heightened survival rates and biomass production, taller plants, increased leaf count and higher levels of pH, bicarbonate, calcium and nitrate. Conversely, PC1 displayed strong negative loadings for aluminium and PTEs, indicating their detrimental effects on plants. PC2 played a minor role (10% variance), with all loadings remaining below 0.54. Overall, the PCA unveiled a clear link between thriving *B. juncea* plants and improved soil pore water quality, characterized by lower acidity, reduced stress from aluminium and PTEs and optimal nutrient availability. These findings are consistent with previous research on the beneficial effects of soil amendments on *B. juncea* growth in contaminated soils (Clemente et al., 2005; Fernández-Caliani et al., 2024; Pérez-Esteban et al., 2014).

In the PCA plot, Technosols S3T2-25 and S3T8-5 clustered closely together, indicating similar effects on both plant response and soil solution chemistry. Conversely, Technosol S3T11-35 displayed a notable separation, reflecting distinctive effects on plant growth (e.g. reduced survival rate and biomass) and pore water parameters (e.g. lower pH and higher toxic elements). The distant positioning of the untreated mine soil (control) from these clusters underscores the effectiveness of Technosol-based treatments in promoting plant growth and modifying soil chemistry.

The successful germination, growth and flowering observed in Technosols S3T2-25 and S3T8-5 provide promising evidence of their potential to overcome growth limitations in mine soils. These technogenic soil blends not only enhance plant survival rates but also support flourishing development under controlled pot experiment conditions. Nevertheless, field trials with a variety of plant species may be necessary to fully validate the impact of Technosol amendments on PTE mobility and bioavailability in the soil–plant system, under realistic environmental conditions.

4 | CONCLUSIONS

This pot-based study used *B. juncea* as a model plant to explore the potential of waste-derived Technosols as a sustainable and multifunctional approach for revegetating AMD-impacted soils. The results

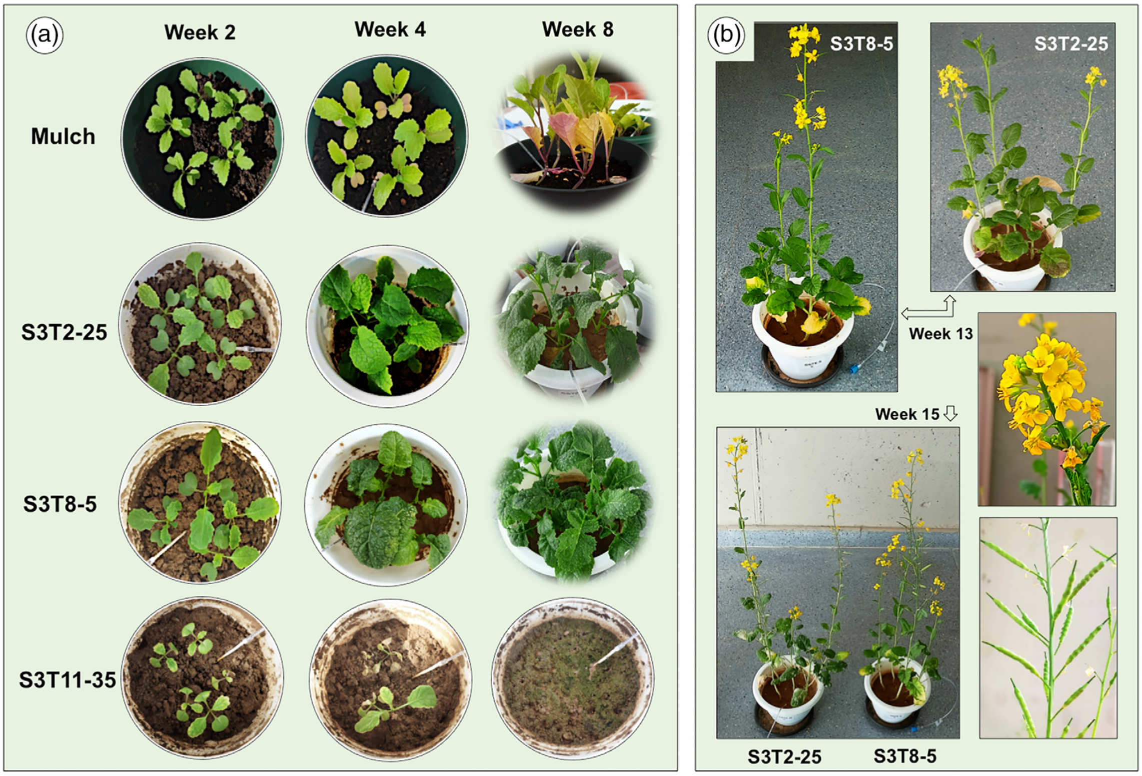
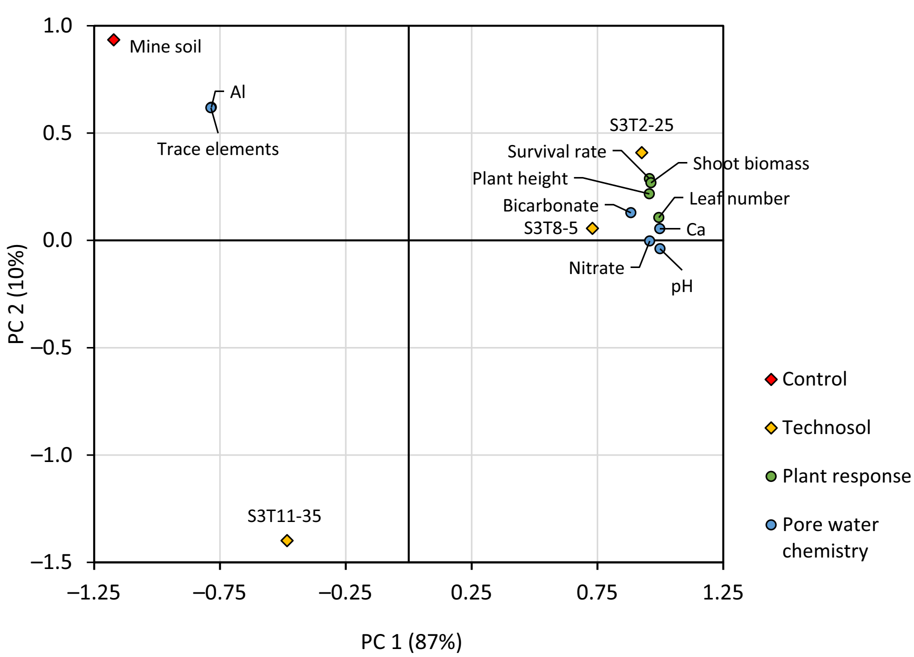


FIGURE 7 Visual comparison of *Brassica juncea* growth in Technosols and mulch control pots (∅ = 15 cm) during the: (a) seedling and early vegetative growth stages; and (b) flowering and fruiting periods. [Colour figure can be viewed at wileyonlinelibrary.com]

FIGURE 8 PCA plot depicting the relationships between plant response parameters and soil pore water chemistry variables in mine soil and Technosols. The percentage of variance explained by each principal component is indicated on the corresponding axis label. [Colour figure can be viewed at wileyonlinelibrary.com]



consistently demonstrated the effectiveness of Technosols in mitigating the extreme acidity of the mine soil, improving nutrient availability and reducing the leaching of PTEs to environmentally sustainable levels. The study identified several concomitant mechanisms for PTE immobilization: reduced dissolution of metal-bearing minerals, precipitation reactions forming stable mineral phases and adsorption onto Fe and Al (oxy)hydroxides. These processes collectively contribute to decrease PTE mobility and bioavailability in the leachate. Aqueous speciation modelling indicated a shift towards less phytotoxic forms of aluminium and heavy metals following treatment. This aligns with the observed germination and robust growth of *B. juncea* in specific Technosol formulations. Technosols S3T2-25 and S3T8-5 supported high survival rates and optimal biomass production, underscoring their capacity to transform barren AMD-impacted landscapes into fertile and revitalized soils. PCA analysis corroborated this positive correlation between improved soil conditions and healthy plant responses. However, Technosol S3T11-35 showed limited effectiveness, emphasizing the importance of carefully selecting and formulating waste amendments to achieve optimal reclamation outcomes. Further research, particularly through field trials, is warranted to confirm these findings and assess the long-term effects of waste amendments on soil quality. This will provide valuable insights for informed decision-making regarding the large-scale implementation of Technosols in real-world settings.

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CONFLICT OF INTEREST STATEMENT

The authors declare no conflict of interest.

DATA AVAILABILITY STATEMENT

The data that support the findings of this study are available from the corresponding author upon reasonable request.

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SUPPORTING INFORMATION

Additional supporting information can be found online in the Supporting Information section at the end of this article.

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